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Carbon Nanotube and Graphene-Based Supercapacitors: Rationale, Status, and Prospects

by Matthew H. Ervin

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In a supercapacitor, the surface area of the electrodes accessible to the solvated electrolyte ions determines the capacitance; therefore, carbon materials with improved surface area may increase the energy density of supercapacitors. Two materials being studied for this are carbon nanotubes (CNTs) and graphene. Since all of their atoms are surface atoms, extremely large surface areas per mass may be obtainable. CNTs also have inherently high conductivity, which should increase the power density of these capacitors. To date, CNT supercapacitors have only matched state-of-the-art activated carbon supercapacitor performance. Factors limiting CNT supercapacitors are explored and future prospects are considered.					
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1. Background

Portable power sources, such as batteries, are a critical part of a Soldier's equipment. Therefore, advances in portable power production and storage are of great interest to the Army.

Supercapacitors have several advantages over conventional batteries, including higher specific power (~2 orders of magnitude higher than batteries and fuel cells), higher cycle life (able to withstand millions of charge/discharge cycles), rapid charge/discharge times (seconds to minutes), high efficiencies (up to 98%), and unaltered performance in extreme heat and cold (1). Their quick-charge capability would mean they could be charged in a few minutes before a patrol. As it is, Soldiers are replacing their batteries before each patrol to guard against equipment failure in the field. This represents a significant logistical burden in supplying and disposing of batteries. Unfortunately, current state-of-the-art supercapacitors lack the energy density to make them competitive with conventional batteries for most portable power applications.

In the area of electrical energy storage, capacitors compete with batteries and fuel cells. (Capacitors have a wide variety of other applications in addition to energy storage, such as providing pulse power and signal filtering, and creating resonant circuits.) Batteries and capacitors are similar in many ways, but they store energy using two different mechanisms. Batteries store energy via reduction and oxidation (redox) reactions at their electrodes, whereas capacitors store energy in an electric field across a dielectric. As a result, batteries and capacitors have significantly different characteristics. Batteries excel at providing high energy densities at low power, while conventional capacitors have high power densities but low energy density. A special type of capacitor, a supercapacitor (also called an ultracapacitor or electrochemical double layer capacitor), has high power density along with moderate energy density.

A traditional capacitor consists of two metal plates separated by a thin dielectric. The larger the overlapping electrode surface area and the thinner the dielectric, the higher the capacitance. An electrolytic capacitor has a metal electrode covered with a dielectric, with the second electrode being a liquid electrolyte. Again, the overlapping surface area between the dielectric-coated electrode and the liquid electrode, along with the dielectric thickness, combine to determine the capacitance.

A supercapacitor consists of two solid electrodes in contact with a liquid electrolyte without any dielectrics (although there is an electrolyte permeable electrode separator to prevent shorting of the electrodes). Supercapacitors store charge by inducing the adsorption of ions onto the electrodes using an electric field. When a potential is applied across the electrodes, the positive ions in the electrolyte will adsorb onto the negative electrode and the negative ions onto the positive electrode. Since there is no dielectric on the electrodes, the applied biases must remain

low enough that charge transfer between the ions and the electrodes, as well as electrochemical breakdown of the electrolyte solvent, do not occur. This limits the voltage rating on individual supercapacitor cells to about 1.2 V when using aqueous electrolytes, and 3.5 V when using organic electrolytes.

2. Rationale for CNTs/Graphene

Supercapacitors were first commercialized in 1978 by NEC Corporation. In a supercapacitor, large capacitances are achieved by using electrodes with very large surface areas. Commercial supercapacitors use carbon electrodes made from highly porous “activated” carbon (typically derived from coconut shells) and a binder material attached to a highly conductive current collector. Carbide-, fiber-, and sugar-derived activated carbons are under development to improve upon the performance of activated carbon. Carbon electrodes are desirable due to their favorable physical and chemical properties. They are conductive, can be made with high surface area, have good corrosion resistance, have good thermal stability, and are made using inexpensive materials (1). Since the surface area of the electrodes accessible to the solvated electrolyte ions determines the capacitance, carbon materials with improved surface area may increase the capacity of supercapacitors. Two materials being studied for this are carbon nanotubes (CNTs) and graphene. CNTs are single or multiple atomic layers of graphite that curve back on themselves to form tubes, while graphene is a single atomic layer of graphite.

CNTs are potential candidates since, in the case of single wall nanotubes (SWCNTs), all of the carbon atoms are surface atoms; consequently, extremely large surface areas may be obtainable if the CNTs can be assembled in a manner that maximizes the surface area by minimizing CNT bundling and optimizing the porosity of the CNT electrode. The outside of SWCNTs have a theoretical surface area of $1320\text{ m}^2/\text{g}$, though the scientific literature typically reports surface areas of $<450\text{ m}^2/\text{g}$ based on nitrogen absorption studies. This indicates that there is significant potential for CNT surface area improvement by optimizing the morphology of the CNT film. CNTs also have inherently high conductivity, and their fibrous nature may eliminate the need for an inert binder so that the resulting electrode may have improved conductivity. This will contribute to a low equivalent series resistance and, therefore, increased power output. On the downside, CNT growth produces a mixture of 2/3 semiconducting and 1/3 metallic SWCNTs, with the semiconducting tubes expected to contribute little to the capacitance due to their low density of states (2). (Our own experiments have yet to demonstrate this.) If it is true that semiconducting tubes do not contribute significantly to capacitance, that would also mean that graphene nanoribbons formed by unzipping CNTs (3), as was originally proposed, would not be a viable approach since all graphene nanoribbons are semiconducting if they are narrow enough. This would not be a problem with larger graphene platelettes. Multiwall CNTs (MWCNTs) tend to be metallic as chances are good that at least one of the nested shells is metallic, but the

semiconducting shells may still be dead weight. Cost is also a significant issue, as SWCNTs can cost ~\$100,000/kg, and MWCNTs can cost ~\$5, 000/kg, while activated carbon costs just ~\$15/kg.

Graphene is an even more recent entry into the supercapacitor arena. Graphene, again, is composed of all surface atoms, but now both sides of the layer are accessible, giving it a theoretical surface area of $2640\text{ m}^2/\text{g}$. While activated carbon is sometimes measured to have similar surface area, this is almost certainly overstated by the measurement technique; in any case, it is clear that all of the measured surface area is not accessible to the electrolyte ions. A significant advantage of graphene over CNTs is that there are a number of methods—e.g., chemical exfoliation—for producing it from graphite. Indeed, commercial scale production of graphene in ton quantities is underway, which should result in much more competitive prices than for CNTs. While graphene has potentially more surface area, its morphology may make it more difficult to produce a porous electrode, as the graphene sheets are likely to stack on top of one another without intervening pore volume for the electrolyte.

3. CNT/Graphene Processing

Both CNT and graphene materials are frequently processed as suspensions/solutions. In order to get stable dispersions in a solvent, the CNTs or graphene are either chemically functionalized and/or dispersed with the aid of a surfactant. Both approaches have their drawbacks. Chemical functionalization, for instance, by adding carboxylic acid groups to the material with the aid of a strong acid, can make the material more soluble in water. However, the addition of these functional groups introduces defects in the lattice structure of the material that can decrease its conductivity. Such functional groups can also represent red-ox groups, which can incorporate pseudocapacitance in the capacitor that may or may not be desirable. (Pseudocapacitance is (dis)charging resulting from redox reactions like a battery, but which occur at a potential dependent rate so that they behave like a capacitance.) While the presence of pseudocapacitance can increase the capacitance, the eventual decomposition of functional groups and impurities can result in gas liberation—e.g., carboxylic acid groups converting to carbon dioxide (CO_2)—which can rupture the device package. Pseudocapacitance derived from incorporated nanoparticles or thin films of selected materials, such as manganese or ruthenium dioxide (MnO_2 or RuO_2), can increase the total capacitance by several times.

Surfactants can be used in place of functionalization to help solubilize CNTs or graphene, but since they are generally nonconductive, themselves, they will impede charge transfer between the nanomaterials. Therefore, further processing may be required to clean them, which may remove the CNTs/graphene from the substrate or alter the electrode morphology (4).

These solution-based processing issues can be avoided by growing the CNTs or graphene directly on the current collector. In the case of CNTs, this has been done using chemical vapor deposition (CVD) by several groups (5). Growing vertical arrays of CNTs is attractive, as the vertical CNTs have, in effect, vertically aligned pores that allow for efficient ion transport into and out of the electrode. On the other hand, it may be difficult to optimize the density (porosity) of the electrode, and the process has limited throughput. As CVD is a high temperature >700 °C process, there are also limits on the current collector materials used, and there is difficulty integrating the process for on-chip or other specialized applications. For these reasons, and increased options for manufacturing, solution-based processing is likely to be a more cost-effective and flexible option. Many solution-based processing approaches have been demonstrated with some recent examples given in table 1. The solution-based processing approaches in table 1 have demonstrated 23–200 F/g, although the higher values likely include pseudocapacitive contributions. For comparison, state-of-the-art activated carbons yield specific capacitances of up to 120 F/g. A review of the scientific literature has shown that commercially available activated carbon supercapacitors using aqueous electrolyte exhibit a specific capacitance of 33 F/g, a specific energy of 13 Wh/kg, and a specific power of 0.58 kW/kg (6).

Table 1. Examples of solution processed CNT/graphene electrodes.

Deposition method	CNT solution	F/g	Comments	Reference
Spray Deposition	SWCNT	38	CNTs not functionalized	7
Spray Deposition	SWCNT-COOH	155	Functionalization results in clear redox pseudocapacitance	7
Spray Deposition	MWCNT	23	CNTs not functionalized	7
Spray Deposition	MWCNT-COOH	77	Functionalization results in clear redox pseudocapacitance	7
Spray Deposition	SWCNT	90–120	CNTs possibly functionalized during purification	8
Layer by Layer	MWCNT-COOH MWCNT-NH ₂	150	Pseudocapacitance contribution	9
Filtration	DWCNT	67	DNA dispersed double walled CNTs	10
Filtration	DWCNT	32	Double walled CNTs	11
Electrospray	MWCNT-COOH	108	Pseudocapacitance contribution	12
Meyer Rod - Adsorb on Paper	SWCNT	200	Measurements indicate at least some pseudocapacitance	4
Freeze Dry	graphene	120	200C anneal of graphene oxide may not remove all oxide functional groups	13

4. Capacitance Limits

What is the theoretical specific capacitance for SWCNTs? The theoretical limits are determined by the surface area per gram, which is $1320\text{ m}^2/\text{g}$ for SWCNTs, and the size of the solvated ions of the electrolyte, which combine to determine how many ions can adsorb on a gram of SWCNTs. The capacitance achievable per electrode area for potassium hydroxide and sulfuric acid (KOH, H_2SO_4), and common organic electrolytes are 74, 42, and $\sim 20\text{ }\mu\text{F}/\text{cm}^2$ (valid at $\sim 1\text{ V}$), respectively. (At very high electrolyte concentrations, these numbers can be increased when the effective ion size is reduced by preventing the outer salvation spheres with a shortage of solvent.) This yields a theoretical specific capacitance limit of 977 F/g for SWCNTs; this amount is double for graphene. If one assumes the usual ratio of 1/3 metallic SWCNTs, and that the semiconducting tubes do not participate, this number is reduced to 325 F/g for SWCNTs.

It should be noted that activated carbon does not come close to its maximum specific capacitance, calculated using nitrogen absorption surface area measurements. This is probably due to errors in the measurement methodology, as well as to the fact that surface area available to nitrogen molecules is not all available to the larger solvated electrolyte ions (14). Perhaps a more important restriction is that as ions are electrostatically drawn into the activated carbon pores, they naturally adsorb at the first available site. Consequently, the mouth of the pore is blocked by the first ions to arrive, leaving the rest of the pore area to be unused (15). Since CNTs and graphene have their surface area all on the outside, it should be possible to tailor their morphology to more effectively use the surface area.

As it turns out, there may be another reason CNTs, graphene, and activated carbon do not attain the theoretical specific capacitances calculated. Single-layer graphene or SWCNTs may not be able to polarize a full monolayer of electrolyte ions due to an insufficient number of carriers in one atomic layer. This implies that there are not enough carriers in a CNT or graphene sheet to completely screen the field from the adsorbed ions so that the field penetrates through to the other side, diminishing the number of ions that can adsorb there (16, 17). Alternately, one can think of the electrode capacitance being a combination of interfacial capacitance (the adsorbed ions/double layer) and the quantum capacitance of the CNT/graphene (18, 19). Since these capacitances combine as capacitors in series, the smaller capacitance (in this case, the quantum capacitance) will dominate so that the calculated interfacial capacitance will not be achieved. If one were to assume that it takes two CNT shells to screen the field from ions adsorbed on the outside, then the maximum specific capacitance would be achieved for double-wall CNTs with 490F/g (in KOH) with all shells metallic, or 160 F/g for mixed semiconducting and metallic shells (if the semiconducting shells do not contribute). Graphene would then need four layers to support the full capacitance available with its surface area, with a similar reduction in achievable specific capacitance. More work is required to determine if this is a serious limitation.

5. Capacitor Evaluation

When characterizing capacitors, one needs to be careful to distinguish between true capacitance and any redox contributions. In table 1, the metric of specific capacitance has been used as a measure of how much useable surface area is being obtained. When pseudocapacitance is present, it can then be difficult to determine how much capacitance is due to true capacitance resulting from the large surface area versus that due to redox-based pseudocapacitance.

However, even without pseudocapacitance, it can be difficult to compare results. Factors such as measurement methodology (cyclic voltammetry versus constant current measurements), measurement parameters, and the electrolytes used can contribute to significant differences in measured capacitances. The measurements must be made under realistic conditions so that measured capacitances will reflect practically usable values. A review of the literature by Istvan has revealed that changing the CV scan rate from 5 to 20 mV/s, or the galvanostatic charge/discharge current from 10 to 200 mA/g, will reduce the measured capacitance by 20% (15). Standard test conditions of 20 mV/s and 200 mA/g or higher are, therefore, recommended. Another important testing factor is that the device must be cycled many times before testing in order to exhaust any irreversible redox contributions to the capacitance due to impurities that would exaggerate the measured capacitance.

To this point in the discussion, I have focused on the specific capacitance (F/g) figure of merit, which reflects how effectively the electrode material is being used. Other important figures of merit include rated voltage (V), capacitance (F), resistance (Ω), time constant (s), specific energy (Wh/kg), and specific power (W/kg), as well as volumetric measures of capacitance, power, and energy. Obviously, many of these factors are interrelated. In research papers, the specific capacitance is usually calculated using the mass of the carbon (e.g., CNTs) only, since it reflects how good the carbon electrode morphology is, and since the electrodes are frequently not packaged into devices. However, in a commercial device, the farads per gram specification will generally be calculated using the entire mass of the capacitor because, for the final application, it is the entire mass of the capacitor that is relevant. In a packaged device, it is the capacitance, energy, and power available per packed device weight or volume that will be most important. To get an idea of where supercapacitor technology stands, it is useful to look at a survey of activated carbon supercapacitors under industrial development, which have been characterized in Andrew Burke's lab at the University of California (UC)-Davis (20). Burke has found that the current crop of supercapacitors under development have specific capacitances of 3 to 10 F/g (total device weight), specific energies of 2.3 to 12 Wh/kg, specific powers of 390–5700 W/kg, and volumetric capacitances of 3–15 F/cc. These are important specifications to keep in mind while developing CNT/graphene supercapacitors.

6. Conclusions and Opportunities

CNTs have yet to convincingly exceed the specific capacitance of activated carbon in the absence of pseudocapacitance, but even if they do not, CNTs/graphene may still yield improvements in power due to increased electrode conductance. There may also be important supercapacitor improvements due to the mechanical properties of CNTs/graphene. For instance, CNTs/graphene may lend themselves to flexible, conformal, or integrated supercapacitors that would be useful for applications where there is little available space. It would also be desirable to develop supercapacitors that could be used as structural elements.

Much recent work on supercapacitors has focused on increasing their energy density so that they can compete with batteries. This may take the form of incorporating increasing amounts of pseudocapacitance. Similarly, a lot of effort has gone into increasing battery power density, which may be achieved by reducing the discharge depth at the expense of energy density. In each case, a technology's strength is being compromised in order to shore up a weakness. In the end, it is probably more logical to develop a hybrid system that couples a supercapacitor to a battery or other energy source. The battery slowly charges the supercapacitor, which then supplies the peak power loads of the system. The supercapacitor could also level the load from intermittent or regenerative power recovery systems. In this approach, the supercapacitor could extend the power load the battery could support and extend the battery's lifetime by reducing the number and depth of charge/discharge cycles. Such a hybrid could capture the best performance of each technology.

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List of Symbols, Abbreviations, and Acronyms

CNT	carbon nanotube
CO ₂	carbon dioxide
CVD	chemical vapor deposition
DWCNT	double wall carbon nanotube
F	farads (capacitance)
F/g	farads per gram (specific capacitance)
H ₂ SO ₄	sulfuric acid
KOH	potassium hydroxide
MWCNT	multi-wall carbon nanotube
MWCNT-COOH	carboxylic acid functionalized multi-wall carbon nanotube
MWCNT-NH ₂	amine functionalized multi-wall carbon nanotube
MnO ₂	manganese dioxide
RuO ₂	ruthenium dioxide
S	seconds
SWCNT	single wall carbon nanotube
SWCNT-COOH	carboxylic acid functionalized single wall carbon nanotube
V	volts
Ω	Ohms (resistance)
W/kg	Watts per kilogram (specific power)
Wh/kg	Watt hours per kilogram (specific energy)

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